



Ultrasonic Velocity of the Analysis of Water Content Influence on Phosphatic Chalk

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<http://doi.org/10.29227/IM-2024-01-03>

Submission date: 12.4.2023 | Review date: 2.5.2023

Abstract

Water content may have a crucial effect on the petrophysical and mechanical properties of high porosity rocks such as chalk. Consequently, the water fluctuations may significantly influence the behaviour of engineering structures that are constructed in chalks. It is the aim of this study to illustrate how the elastic and strength properties of chalk can evolve considering water content variation using P-wave velocity and analytical analyses. To study the influence of water on the ultrasonic compressional wave velocity (VP), measurements of travel time of three samples subjected to different water content were performed. The Uniaxial Compressive Strength (UCS) in dry and saturated chalk specimens was also estimated. To clarify the possible influence of lithology on the physicomaterial properties of rocks, three samples of phosphatic chalk were studied in thin sections. Automated mineral analysis and textural imaging of the samples were performed using an FEI QEMSCAN®. The analyses of the Ultrasonic tests reveal that changes in the water content are associated with variations in the P-wave velocity. Based on P-wave velocity changes as a function of water content, analytical models have been used to predict the elastic and strength properties. The data indicate that the presence of water significantly reduced the elastic and strength parameters of the chalk, a result that is in agreement with the UCS laboratory tests. The derived equations can be used for predicting the elastic and strength properties of high-porous chalk from the P-wave velocity as a function of water content. This approach may avoid the necessity for time-consuming laboratory testing.

Keywords: ultrasonic wave velocity, water content influence, phosphatic chalk, uniaxial compressive

Introduction

The weakening effect of water on highly porous rock is a well-known problem in the field of geotechnics and mining. This phenomenon can give rise to a number of issues related to geohazards, ground surface stability, and safety. The presence of water in underground cavities, such as mines, can often be the cause of continuous deterioration of the rock mass properties. In addition, rock masses can undergo significant mechanical changes when subjected to variations in water content due to fluctuations in the groundwater levels. This can result in changes in the structure of the rock mass and behaviour, both in saturation and drying processes (Zhou et al, 2016), leading to some structural changes and instabilities. Therefore, the effect of water content on porous rocks, such as chalk, must be taken into account and analysed when such conditions are present.

Ultrasonic pulse velocity is one of the most widely used non-destructive techniques applied for the assessment of the mechanical properties of rock and concrete materials (Yilmaz et al. 2014). The velocity is strongly influenced by the petrophysical parameters of the rock mass (Kahraman 2007, Descamps et al. 2017). In this test, longitudinal waves are transmitted through rock core samples and the wave propagation velocity is noted and used for analysis. When the P-wave velocity is correlated with the elastic and strength properties of the material under test, these properties can be derived in a non-destructive and fast by applying the ultrasonic test.

Geological and hydrogeological context

The abandoned Malogne phosphatic chalk quarry is located in the central part of the Mons sedimentary basin, western Belgium. The Mons Basin is formed by Cretaceous, Paleogene, and Quaternary formations that unconformably overlie the intensively folded Devonian to Upper Carboniferous basement. In the Upper Cretaceous sequence, where the Malogne phosphatic chalk quarry is located, carbonate and mixed carbonate-siliciclastic rocks prevail. The chalk, represented by 8 lithostratigraphic units that form the Chalk Group, is the most common lithotype for the Campanian to the Maastrichtian stage (Robaszynski et al., 1987, 2001; Kennedy, 1993). The Upper Cretaceous chalk in the Mons Basin contain phosphate components, commonly

represented as beds or lenses of phosphatised chalk lag granules and pebbles, rarer nodules. Mainly in the uppermost part of the section, they come as grains within the chalk matrix becoming a rock-forming component. The Ciply-Malogne Phosphatic Chalk Formation, mined in the Malogne phosphatic chalk quarry, occupies the uppermost (Early Maastrichtian) part of the Mesozoic succession forming a northward dipping lens-like body in a southern flank of the basin.

The rocks of the Chalk Group of the Mons Basin form a regional aquifer system. On a local scale, in the NE parts of the Malogne phosphatic chalk quarry, an aquifer is identified. A hydrogeological monitoring program in the quarry shows that there is a seasonal groundwater-level fluctuation in the Malogne phosphatic chalk quarry. The yearly fluctuation range is within 1 m with a minimum during the winter and a maximum in the summer. Because of the seasonal fluctuation of the groundwater level in the quarry, a part of the pillars is exposed to cycles of water-saturating and drying throughout the year. In terms of the groundwater level position, three different areas can be identified in the site - dry, transitional, and flooded zones (Fig. 1). Due to the general beds dipping to the North, the remaining one-third of the quarry is permanently below the groundwater level. According to Pacyna (1992) and Funcken & Welter (1996), the variation of the groundwater level would be related directly to the stability of the quarry, and therefore needs further investigation.

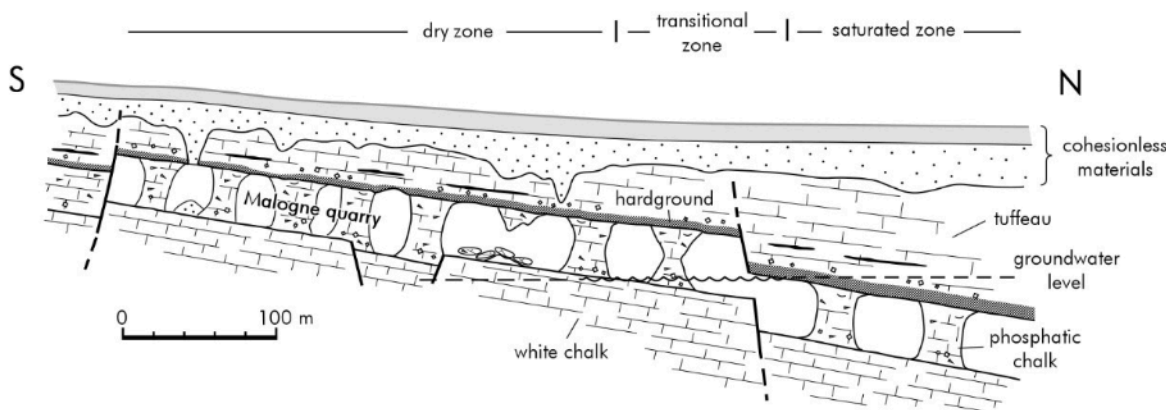


Fig. 1. Schematic cross-section (not scaled) of the Malogne phosphatic chalk quarry (modified after Pacyna, 1992) with the area of the dry, transitional, and water-saturated zones (after Georgieva et al., 2023).

Material and Methods

To better understand the characteristics of the phosphatic chalk, an integrated lithological, petrophysical, and geomechanical study was carried out. For that purpose, samples were taken from the Malogne Phosphatic chalk quarry, Upper Cretaceous chalk, Mons Basin. At first, lithological characterization of the tested material was conducted aiming to determine the chemical, mineral, and textural features of the chalk as well as to characterize the porosity in terms of size and volume. Later, these were correlated with the petrophysical and mechanical properties of the chalk. For that purpose, cylinders with diameter and height of 40 mm and 80 mm, respectively, were prepared and their dry and saturated density and porosity were determined. To study the influence of water on the ultrasonic compressional-wave velocity (V_p), measurements of travel time of three samples subjected to water-saturation-drying cycles, thus experiencing different water content, were performed. In addition, the elastic and mechanical properties of another 26 cylindrical samples from the phosphatic chalk were determined in dry (14 samples) and fully saturated (12 samples) conditions. Lastly, by conducting regression analyses a correlation between the P-wave velocity and the elastic and strength properties were determined and correlation coefficient was determined.

Lithological characterization

On three samples, a complex of automated chemical, mineral, and textural analyses was performed using an FEI QEMSCAN® Quanta 650F at the Department of Earth Sciences, University of Geneva, Switzerland. Analyses were conducted under a high vacuum, accelerating voltage of 25 kV, and a beam current of 10 nA on carbon-coated polished thin sections. In total, 221 individual fields were measured per sample, with 1500 μm per field, and point spacing of 5 μm . The standard 1000 counts per point were acquired, yielding a limit of detection of approximately 2 wt % per element for mineral classifications. Measurements were performed using iMeasure v5.3.2 software and data processing using the iDiscover® v5.3.2 software package. Final results consist of: i) high-quality spatially resolved and fully quantified mineralogical maps; ii) back-scattered electrons (BSE) images with identical resolution as the mineralogical maps; iii) X-ray element distribution maps. For the textural analysis standard optical cathodoluminescence (CL) microscopy was used.

The rock porosity is characterized by volume, type (after Choquette & Pray, 1970), and size (after Loucks et al., 2012).

Petrophysical and geomechanical characterisation

The porosity of all rock specimens was estimated with water according to the American Petroleum Institute (API) standard procedures (API, 1998), as its values could significantly influence the mechanical properties of the intact rock (Ramos da Silva et al., 2010; Palchik & Hatzor, 2004; Descamps et al., 2012).

The P-wave velocity was measured on 50 cylinders in saturated state and on 16 cylinders in dry conditions by Portable Ultrasonic Nondestructive Digital Indicating Tester (PUNDIT) Plus system with 54 kHz P-wave transducers. As a coupling fluid was applied vaseline on the end surface of the samples and transducers (transmitter and receiver).

Based on the measured by the direct transmission technique time of ultrasonic pulses propagation (precision of 0.1 μs) and length (precision of 0.01 mm) of the samples. Based on the estimated velocity (Rummel and van Heerden, 1978) the preliminary elastic properties of the studied rock are determined.

The P-wave velocity changing as a function of water content was studied. Concerning the drying cycles, a free drying of the samples was performed. For each sample, such saturation-drying cycle was performed three times.

In addition to the dry- and wet-rock P-wave velocity measurements on 50 samples, from them, 14 dry samples and 12 saturated samples were tested under uniaxial compression and their UCS was determined.

Results and Discussion

Lithological characterization

Generally, the studied samples are relatively weakly lithified, friable, light-colored limestones that represent peloidal to bioclastic wacke- to packstones (Fig. 2). From the mineralogical point of view, the phosphatic chalk is build up mainly by calcite and apatite (Fig. 2d). The calcite dominates (52-55 vol. % of total grains) and is represented mainly by microns in size plankton skeletons (coccolithophores) and less by larger fragments of foraminiferal tests, bivalve shells, ostracod, crinoids, etc. The apatite is between 42 and 44 vol. % and is presented by peloids - mainly fecal pellet, less intraclasts, and rare probably detrital grains (Fig. 2c). Quartz occurs mainly as silt-size detrital grains. The total amount of the other detrital minerals as feldspars, biotite, amphibole, garnets, rutile, ilmenite, sphene, and cassiterite is less than 0.5 %. Clay minerals represent a very restricted amount of kaolinite. Dolomite is not recognized. The cement is predominantly calcite, insignificant in presence, and represented by only one generation (Fig. 2b).

The average volume of the porosity in the studied samples ranges from over 30 % (Fig. 2d), reaching in one of the samples to 40.1 (Fig. 2c). Two main types of porosity - intra- and intergranular, are widely recognized (Fig. 2b-c). Intragranular porosity is mostly related to the foraminiferal tests and is typical of the phosphatic chalk. In addition, the intergranular porosity is well presented. Both meso- and microporosity are typical for phosphatic chalk samples.

As the origin, the porosity is predominantly a primary (syndimentary) porosity, with a minor effect of secondary porosity due to the dissolution of crinoid prisms and aragonite bioclasts.

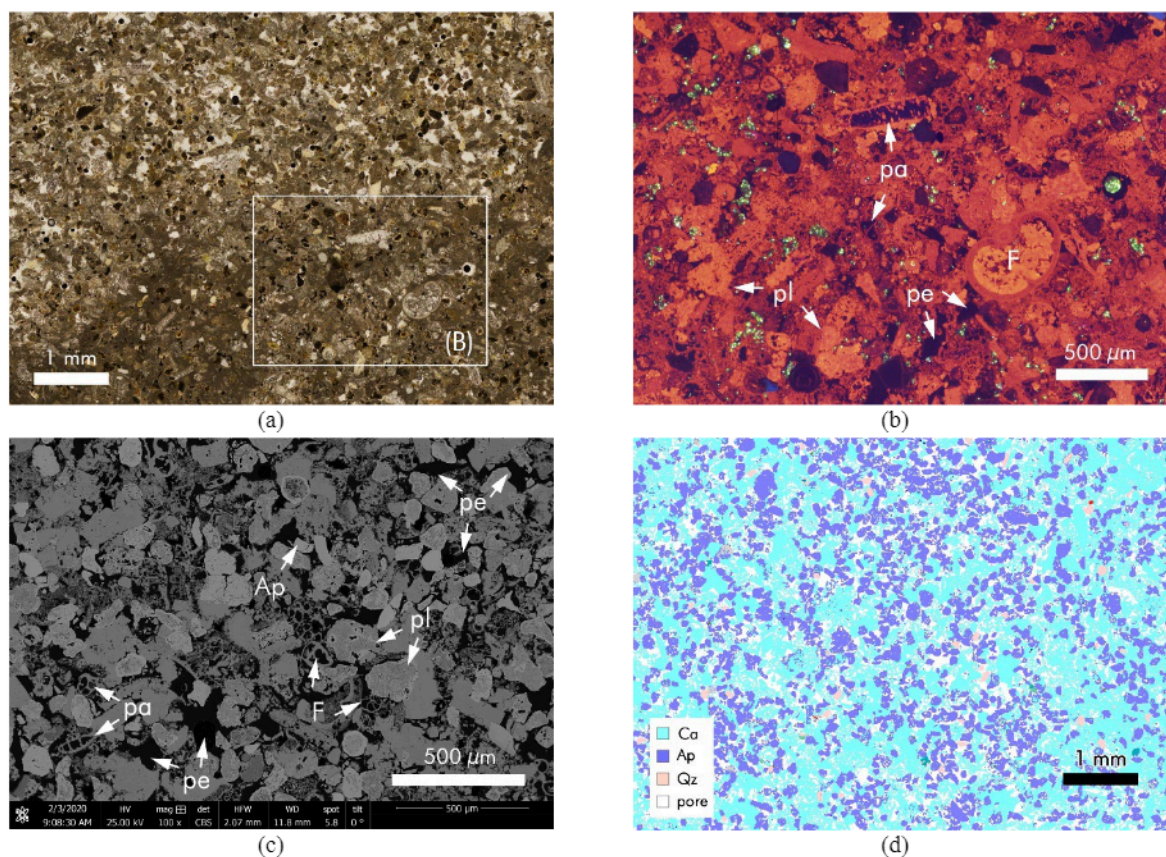


Fig. 2. Lithological characterization of the studied phosphatic chalk samples: (a) transmitted light microscope images; (b) cathodoluminescence (CL) photomicrography of the area marked in photo (a); (c) back-scattered electrons (BSE) images; (d) mineral map based on QEMSCAN study. Abbreviations: Ap- apatite; F - foraminifera; pa - intragranular porosity; pe - intergranular porosity; pl - pellets.

P-wave velocity

The P-wave velocity data for both dry and saturated samples of the phosphatic chalk ranges between 1990 m/s and 2940 m/s (Tabl. 1).

It is considered that the porosity of the chalks is the main control of the ultrasonic compressional-wave velocity in them, such that when porosity increases, the P-wave velocity decreases (Borre, 1998; Mavko et al., 2009). The data obtained in this study confirm well such a relation (Fig. 3). A linear negative correlation between the P-wave velocity and the porosity both in dry and saturated conditions was found. With the exception of a few saturated samples, where relatively higher velocity was measured compared to all other samples, the data vary to a relatively limited extent. Nevertheless, the dry sample results for the P-wave velocity are somehow more scattered and show a lower coefficient of determination than the saturated ones.

The effect of the water content on the P-wave velocity values is the other important focus of the performed ultrasonic tests. The different water content influence on the ultrasonic compressional-wave velocity was further studied in three phosphatic chalk

samples. The specimens were water-saturated and free dried along with measurements of the P-wave velocity until no changes in the sample weight were observed.

Tab. 1. Summary of data obtained by measurements on intact phosphatic chalk samples.

Sample state	Value	P-wave velocity	UCS	E
		m/s	MPa	MPa
Dry	min	1990	5.9	1670
	max	2440	11.2	2770
	avg.	2230	7.9	2100
	st. dev.	0.10	1.4	430
Water-saturated	min	2120	3.0	600
	max	2940	4.6	1670
	avg.	2390	4.0	1160
	st. dev.	0.15	0.47	271

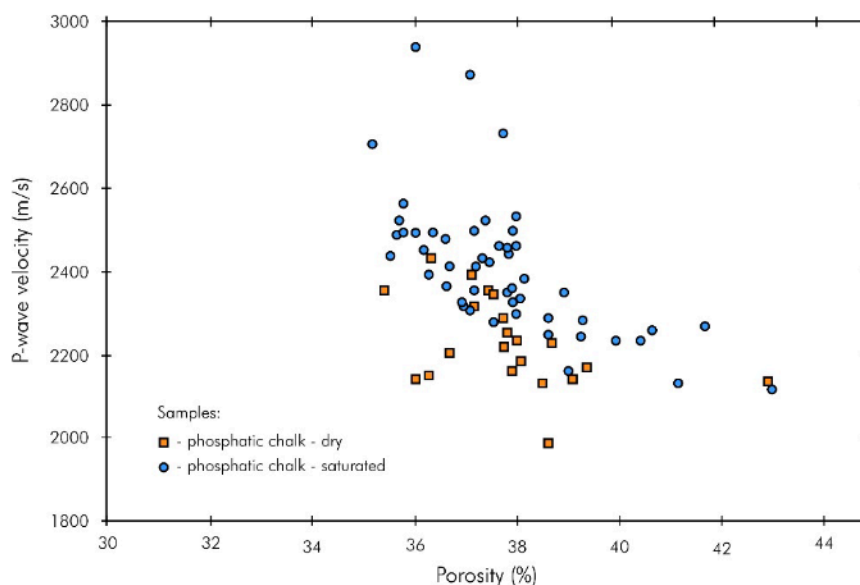


Fig. 3. Relationship between P-wave velocity and porosity measured in dry (orange points) and water-saturated (blue points) phosphatic chalk samples.

This procedure has been repeated three times for the same cylindrical samples, which has resulted in similar observations confirming the repeatability of the measurements. However, it was observed that the first saturation-drying cycle presents more consistent results compared with the second and the third cycles. Therefore, the further analyses are focused on the first cycle of water-saturation and free-drying. The P-wave velocity data for every sample is plotted as a function of water content (Fig. 4).

Generally, for all the samples, the initial increase in the water content is associated exponentially with a decrease of the P-wave velocity. This trend is observed until a water content of approximately 76 % for the phosphatic chalk, from which point afterward the P-wave velocity tends to increase. Thus, two intervals can be identified, one of P-wave velocity decrease where the water content varies between 0 % and 76 % and a second interval in which an increase in the velocity occurs while the water content is also increasing from about 76 % to 100 %. It must be noted that the water distribution in the specimens was not controlled during the experiments, which could contribute to the P-wave velocity and path. The highest P-wave velocity values were measured for the chalk samples in the dry condition. The same results were reported for the Lincolnshire chalk by Sadeghi and Khosravi (2003).

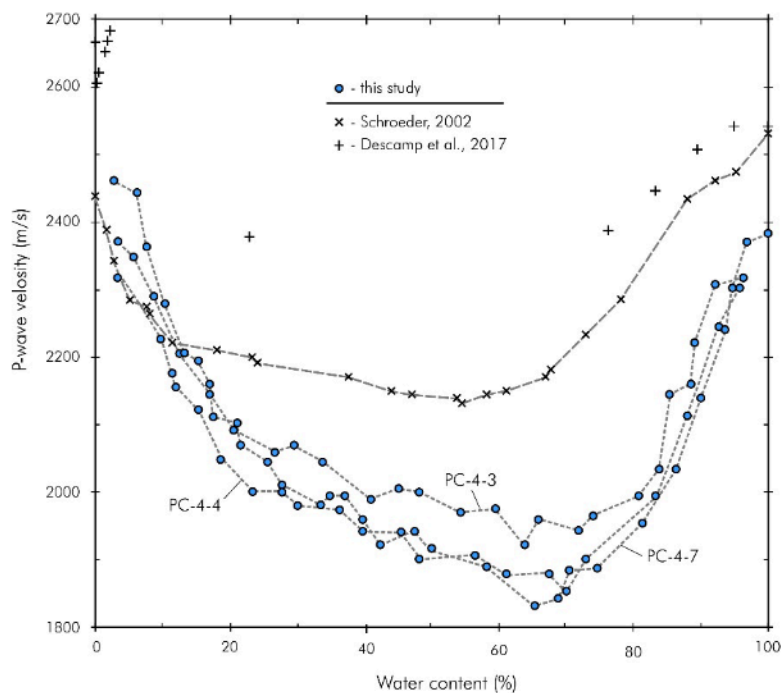


Fig. 4. Evolution of the P-wave velocity as a function of the water content, measured in phosphatic chalk samples, and compared with those published by Schroeder (2002) and Descamps et al. (2017) identical data.

Similar trends have been reported by Schroeder (2002). Studying Campanian chalks in Belgium the same author reported a steady decrease trend in the sonic velocity as a function of water-saturation until the saturation reaches 60 %. Further increasing of sample saturation leads to sonic velocity rise until a maximum value is measured for the complete water-saturated chalk sample. According to Gregory (1976), such a decrease in sonic velocity between 10 % and 60 % saturation degree is difficult to explain and does not correspond to the data in the literature for rocks other than chalks. Comparable results for the evolution of P-wave velocity with water-saturation have been also reported by Descamps et al. (2017). In this case, the P-wave velocity was at a maximum for dry rocks and rapidly decreased for small water-saturation.

The observed trends in P-wave velocity evolution as a function of the water content could be explained by the fact that with sample saturation initiation the pore pressure increases that reduce the P-wave velocity and thus, P-waves travel at a lower speed in water (Descamps et al., 2017). According to other authors (e.g. Gassmann, 1976; Bourbié et al., 1986), the introduction of water to a dry sample increases its density and thus results in a decrease in the velocity. Further, with increasing the water content, the apparent rigidity of the material, as well as the velocity, decreases. Beyond a limit of saturation, water compressibility becomes significant and tends to harden the material, as predicted by Gassmann (1976).

Elastic and strength properties

The Unconfined compression tests were performed through a manual loading rate applied on a stiff frame with a maximum capacity of 20 kN. The load was performed continuously and pressure transducers (350 bar) were used to measure the axial stress. Axial and lateral displacement transducers were used for recording the displacements for each sample. Stress-strain curves for phosphatic chalk were drawn (Georgieva et al, 2020). Based on the stress-strain curves obtained from the UCS tests, it can be concluded that in an atmospheric condition, the tested materials exhibit elastoplastic behaviour with brittle failure.

In dry conditions, the compressive strength of the phosphatic chalk varies from 6.3 to 8.9 MPa (on average 7.8 MPa) and can be characterized as low (Tabl. 1). In a water-saturated state, it decreases approximately by half and is defined as a very low ranging from 3.2 MPa to 4.6 MPa and an average value of 4 MPa. As the strength parameters are typically closely dependent on the porosity, a relationship between porosity and UCS is established (Fig. 5a). A clear trend is observed for the dry state samples where in general higher UCS values are associated with lower chalk porosity and the reverse, the high porosity samples tend to exhibit lower UCS values. In contrast, the water-saturated samples show no clear trend in terms of correlation with the porosity values (Fig. 5a). However, the majority of the tested sample demonstrates that the higher UCS values are obtained from samples with lower porosity.

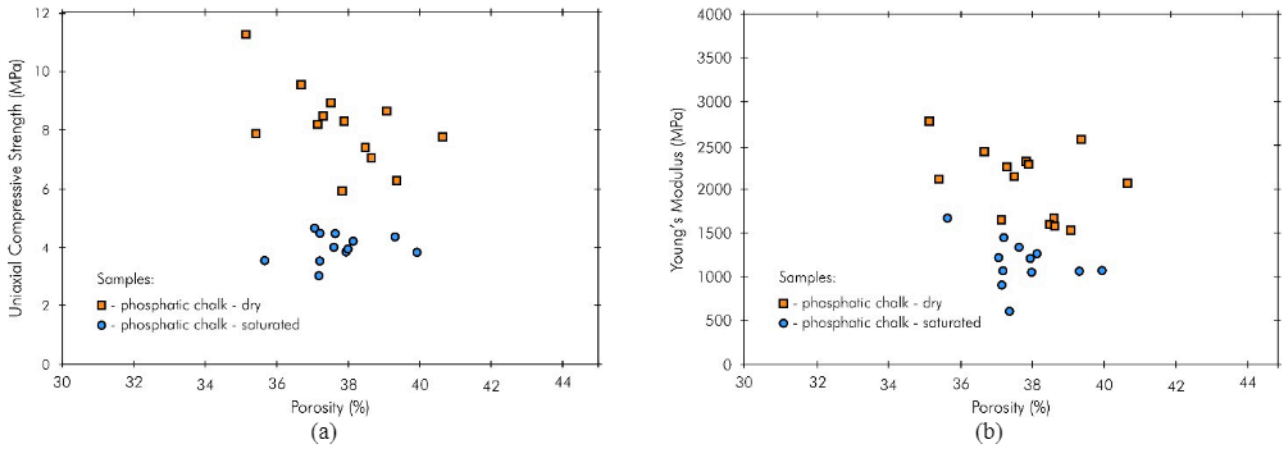


Fig. 5. Relationship between (a) UCS and porosity and (b) Young's Modulus and porosity measured in dry (orange points) and water-saturated (blue points) phosphatic chalk samples.

Another property estimated as the slope of the linear part of the stress-strain curves is the Young's modulus. Its values vary between 1670 MPa and 2770 MPa with an average value of 2100 MPa in dry conditions. When samples are tested in the water-saturated condition a significant decrease of the Young's modulus is obtained ranging from 600 MPa to 1670 MPa. When analysed as a function of the porosity, the general trend of Young's modulus results shows a decrease with porosity increases. However, data for both the dry and saturated samples are rather scattered. Nevertheless, it is clearly demonstrated the saturated samples have lower Young's modulus than the dry ones.

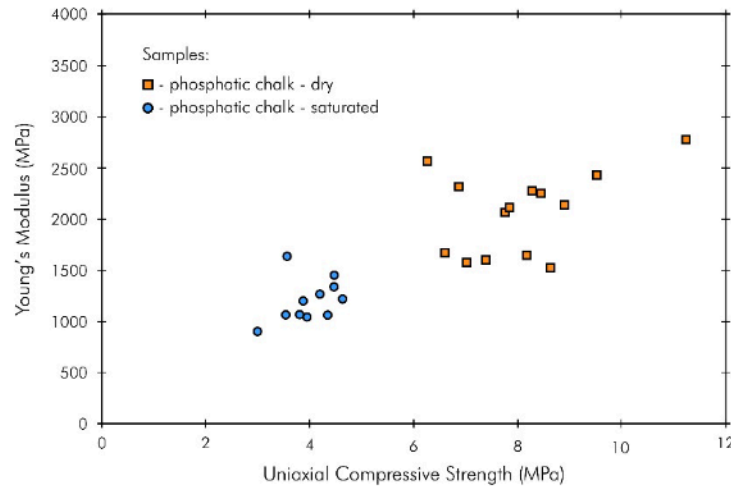


Fig. 6. Relationship between Young's Modulus and Uniaxial Compressive Strength measured in dry (orange points) and water-saturated (blue points) phosphatic chalk samples.

Regression analyses

As seen from Figure 2, P-wave propagation velocity tends to exponentially decrease with water content increase until reaching a water content of 76 %. Therefore, the rest of the analysis will be focused on first cycle results with water content between 0 % and 76 % (Figure 7a).

Further on, a non-linear regression was performed to obtain the mathematical model between V_p and water content (WC) using the non-linear least squares method implemented in Scipy (Vitanen et al, 2020). From the curve fitting, the following relation was obtained (Figure 3):

$$v_p = 1.872 + 0.591e^{-0.057WC} \quad (1)$$

With a coefficient of determination of 0.8535, RMSE = 0.05423, and the standard deviations of the obtained correlation parameters are 9.39×10^{-5} , 0.00037 and 1.87×10^{-5} , respectively.

The residuals of the regression analyses demonstrate an approximately constant variance and zero mean that leads to homoscedasticity, proving the quality of the fit. Figure 7a presents the graphical representation of the obtained equation for water content between 0 and 76%.

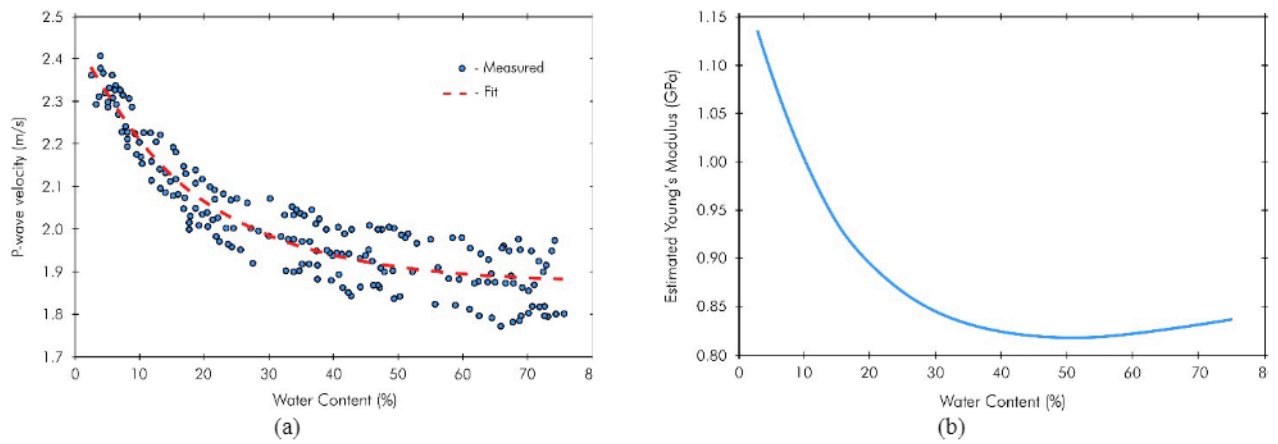


Fig. 7. Non-linear regression from the ultrasonic data from the first drying cycle (red line) for water content between 0 and 76 % (a) and estimated Young's Modulus as a function of the water content (b).

In the same way, curve fitting by non-linear least squares was performed to obtain the mathematical relation between dynamic Young's modulus, determined considering one-dimensional P-wave propagation (Kolsky, 1964). For this purpose, it is also needed to determine the relation between density (ρ) and water content (WC) and the correspondent fitting offers the relation obtained in Equation 2.

$$\rho = 1627.30 + 3.841WC \quad (2)$$

With a coefficient of determination (R^2) of 0.9551.

Also, from the results obtained from the UCS tests with the determination of Young's modulus, the following correlation was obtained:

$$E = 0,1233E_{dynamic} \quad (3)$$

With a coefficient of determination of 0.8574, where E (in GPa) represents the static Young's modulus and $E_{dynamic}$ (in GPa) the dynamic Young's modulus considering one-dimensional P-wave propagation. With these results it is possible to compute the relation between E and water content (Figure 5) considering the general Equation 3. The correspondent graphical representation is presented on Figure 7b.

$$E = \frac{0,1233}{1 \times 10^9} \times [(1.872 + 0.591e^{-0.057WC}) \times 1000]^2 \times (1627.304 + 3.841WC) \quad (4)$$

Finally, knowing the widely used relation between UCS and P-wave propagation velocity.

$$UCS = 2.3547v_p \quad (5)$$

or,

$$UCS = 2.3547 \times (1.872 + 0.591e^{-0.057WC}) \quad (6)$$

With $R^2 = 0.8365$. In Equation 5, UCS corresponds to the uniaxial compressive strength (in MPa) and v_p the P-wave propagation velocity (in km/s).

Conclusion

Ultrasonic tests were conducted on three samples subjected to different water content in saturation - free drying cycles in order to investigate the effect of water on the elastic and strength properties of a phosphatic chalk. Two intervals can be identified in relation to the water content. The first one, in which the water content is ranging between 0 % and 76 %, as the water content increase the P-wave velocity decrease. In this interval, initially a rapid reduction of the P-wave velocity occur at relatively low water content. Afterwards the effect is less pronounced showing moderate velocity decrease. In contrast, in the second interval an increase of the P-wave velocity starts once the water content is higher than 76 %. Nevertheless, the highest velocity is measured when the samples are in fully dry condition. The high primary porosity and relatively weak lithification of the phosphatic chalk contribute to the observed measurements of the P-wave velocity.

To characterise the phosphatic chalk in the two extreme condition, namely dry and fully saturated, geomechanical tests were performed in these conditions. The mechanical behaviour of the phosphatic chalk resulted to be strongly affected by the presence of water in the pores, with strength and stiffness that significantly reduce in the transition from dry to saturated conditions. Similar substantial reduction in properties can be of concern when the high porous chalk is forming the pillars in an underground mine as this can have a direct effect on the stability of the mine. Therefore, quick and reliable approach for estimation of the material properties could be of interest.

Considering the laboratory tests for the determination of the ultrasonic velocity in phosphatic chalk samples with different water contents, a correlation to the correspondent elastic and strength properties was deduced. The obtained results successfully

lead to the determination of the non-linear mathematical relation between the water content and the ultrasonic velocity, with a coefficient of determination of 0.85. Finally, given the determined elastic relations, it was possible to deduct the relation between water content and the strength of the studied phosphatic chalk.

It is, therefore, advantageous and reliable to predict the effect of water on the elastic and strength properties of the chalk in response to the water content variation based on non-destructive and inexpensive techniques such as the Ultrasonic P-wave velocity.

Acknowledgments

This work has been achieved in the framework of the Interreg FWVL RISSC project (2018-2022), with the financial support of the European Regional Development Fund and Wallonia.

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